भारतीय मानक
Indian Standard

IS 8249 : 2019

जिंक सल्फेट हेप्टाहाइड्रेड कृषि ग्रेड — विशिष्टि

(दूसरा पुनरीक्षण)

Zinc Sulphate Heptahydrate, Agricultural Grade — Specification

(Second Revision)

ICS 65.080

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली-110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.org.in www.standardsbis.in

May 2019 **Price Group 6**

Soil Quality and Fertilizers Sectional Committee, FAD 07

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Soil Quality and Fertilizers Sectional Committee had been approved by the Food and Agriculture Division Council.

In the first revision of this standard which was taken up in 1994, consideration was given to the need for maintaining co-ordination with the specifications of the *Fertilizer (Control) Order*, 1957 and the *Essential Commodities Act,* 1955 of Government of India. Consequently, three methods of test for determination of zinc were included, out of which method 3 was described as referee method in case of any dispute.

The present revision is taken up to align it with the requirements of *Fertilizer (Control) Order,* 1985. The following changes have been effected in this revision:

a) The amendments issued to the previous revision have been incorporated.
b) Limits for cadmium, arsenic and sulphur and their methods of tests have been incorporated.
c) The packaging requirements have been modified.

In the formulation of this standard, due consideration has been given to the provisions of the *Fertilizer* (*Control) Order*, 1985, the *Essential Commodities Act*, 1955 and the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011. However, this standard is subject to the restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ZINC SULPHATE HEPTAHYDRATE, AGRICULTURAL GRADE — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for zinc sulphate heptahydrate, agricultural grade.

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
2711 : 1979	Direct reading *p*H metres (*second revision*)
5985 : 1985	Code of practice for handling and storage of bagged fertilizers (*first revision*)
6092	Methods of sampling and test for fertilizers:
(Part 1) : 1985	Sampling
(Part 5) : 1985	Determination of secondary elements and micronutrients (*first revision*)
(Part 6) : 1985	Determination of moisture and impurities
7017 : 1973	Method of colorimetric determination of traces of heavy metals by dithizone
7212 : 1974	Methods of determination of copper
7406 (Part 1) : 1984	Specification for jute bags for packing fertilizers: Part 1 Laminated bags manufactured from 407g/m^2: 85 × 39 Tarpaulin fabric
9755 : 2016	High Density Polyethylene (HDPE)/Polypropylene (PP) woven sacks for packing fertilizers — Specification (*fifth revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of crystals consisting essentially of $ZnSO_4.7H_2O$.

3.2 The material shall also comply with the requirement given in Table 1 when tested in accordance with methods prescribed in col 4.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in laminated jute bags

Table 1 Requirements for Zinc Sulphate Heptahydrate, Agricultural Grade
(*Clauses* 3.2 *and* 6.2)

Sl No.	Characteristic	Requirement	Methods of Test, Ref to Clause
(1)	(2)	(3)	(4)
i)	Matter insoluble in water, percent by weight, *Max*	1.0	Annex F
ii)	Zinc (as Zn), percent by weight, *Min*[1)]	21.0	Annex A
iii)	Lead (as Pb), percent by weight, *Max*	0.003	Annex D
iv)	Copper (as Cu), percent by weight, *Max*	0.1	Annex C
v)	Magnesium (as Mg), percent by weight, *Max*	0.5	Annex B
vi)	*p*H (5 percent solution) not less than	4.0	Annex E
vii)	Sulphur (as S), percent by weight, *Min*[1)]	10.0	**5.3** of IS 6092 (Part 5)
viii)	Cadmium (as Cd), percent by weight, *Max*	0.002 5	Annex G
ix)	Arsenic (as As), percent by weight, *Max*	0.01	**5.3** of IS 6092 (Part 6)

[1)]A tolerance of 0.2 units of nutrient shall be permissible.

conforming to IS 7406 (Part 1) or high density polyethylene (HDPE)/Polypropylene (PP) woven sacks conforming to IS 9755, in quantities as stipulated in *Essential Commodities Act*, 1955 and the *Legal Metrology (Packaged Commodities) Rules*, 2011 and in accordance with *Fertilizer (Control) Order*, 1985.

4.2 Marking

The containers shall be securely closed and marked with the following:

a) Name of manufacturer/Pool handling agency/ Importer (where a manufacturer is also pool handling agency, word 'P' and as the case may be, if an importer the word 'I' shall be written against the name of such manufacturer, if the bag contains imported fertilizer);
b) Manufacturing/Registration Certificate Number
c) Trade mark and/or Brand name, if any;
d) Name of the fertilizer (in case of imported fertilizer, the word 'Imported' shall be super scribed);
e) Percent nutrient as Zn;
f) Gross and net quantity in kilogram;
g) Batch number;
h) Month and year of manufacture/import (in case of imported fertilizer); and
j) Any other information required under the *Fertilizer (Control) Order*, 1985 and the *Legal Metrology (Packaged Commodities) Rules*, 2011.

4.2.1 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder. and the products may be marked with the standard mark.

5 HANDLING AND STORAGE

5.1 The handling and storage of the material shall be in accordance with IS 5985.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 6092 (Part 1).

6.2 Number of Tests and Criteria for Conformity

6.2.1 Zinc shall be tested on each of the individual samples.

6.2.2 The remaining characteristics given in Table 1 shall be tested on the composite sample.

6.2.3 The lot shall be considered to have satisfied the requirement for zinc, if test results on each of the individual samples meet the corresponding requirement given in Table 1.

6.2.4 The lot shall be considered to have met the remaining requirements given in Table 1, if each of test results on the composite sample satisfies the corresponding requirement given in Table 1.

6.3 The lot shall be declared as conforming to the requirements of the specification if **6.2.3** and **6.2.4** are satisfied.

7 TEST METHODS

7.1 Test for requirments listed under **3** shall be carried according to method prescribed in col 4 of Table 1.

8 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which suffer the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF ZINC

Three methods have been prescribed for determination of zinc sulphate heptahydrate, agriculture grade. All the methods can be used for determination of zinc on routine basis. However, in the event of any dispute, Method 3 should be treated as a reference method.

Method 1

A-1 ETHYLENE DIAMINE TETRAACETATE (EDTA) METHOD

A-1.0 Outline of the Method

Zinc is titrated against ethylene diamine tetra acetate (EDTA) solution. The initial separation of zinc from impurities is done by zinc sulphate precipitation.

A-1.1 Reagents

A.1.1.1 *Dilute Sulphuric Acid* — (1:100)

A-1.1.2 *Ammonium Sulphate* — AR grade.

A-1.1.3 *Hydrochloric Acid* — (1:1).

A-1.1.4 *Sodium Hydroxide Solution* — 1 N.

A-1.1.5 *Methyl Orange Indicator* — 0.05 percent.

A-1.1.6 *Methyl Red Indicator*

Dissolve 0.1 g of methyl red in 50 ml of ethanol and dilute to 100 ml with water.

A-1.1.7 *Hydrogen sulphide generator*

A-1.1.8 *Ethylene Diamine Tetra Acetate (EDTA) Solution* — 0.01 M

Dissolve 3.723 g of ethylene diamine tetra acetate dehydrate in distilled water and make up the volume to 1 litre in a 1 litre volumetric flask

A-1.1.9 *Zinc Ion Solution* — 0.1 M.

Weigh about 1.63 g of zinc shot or zinc metal (of 99.9 percent purity) reagent grade accurately. Dissolve in 20 ml hydrochloric acid (1:1), keep it for few hours and allow it to dissolve completely.

Neutralize the resulting solution with sodium hydroxide solution using methyl red indicator. Make up the volume of the solution to exactly 250 ml.

A-1.1.10 *Buffer Solution — p*H 10.

Add 142 ml of concentrated ammonia solution (relative density 0.88 to 0.90) to 17.5 g of ammonium chloride AR grade and dilute to 250 ml with distilled water.

A-1.1.11 *Eriochrome Black T Indicator*

Dissolve 0.2 g of dry stuff in 15 ml of triethanolamine and 5 ml of absolute ethanol.

A-1.2.1 *Standardization of EDTA Solution*

Dilute 25 ml of standard zinc ion solution to 100 ml with distilled water, add 2 ml of the buffer solution and a few drops of the eriochrome black T indicator. Titrate with EDTA solution until the colour changes from wine red to blue.

Note the volume of EDTA consumed as V_1 ml.

Calculate 1 ml EDTA equivalent to zinc as given below:

1 ml of EDTA

$$M_1 = \frac{\text{Mass of zinc weighed in } \mathbf{A-1.1.9} \text{ in mg}}{(0.01\,\text{M})\,\text{solution}\,10 \times V_1}$$

A-1.2.2 Weigh accurately 7.5 g of the sample and dissolve it in water. Make it up to 250 ml. Pipette out 25 ml of the made up solution in a conical flask. Add 6 to 8 g of ammonium sulphate, stir until dissolved and acidify with dilute sulphuric acid adding 1 to 2 drops of methyl orange indicator. Fit the conical flask with a two holes rubber stopper carrying an inlet tube extending to the bottom of the flask and an outlet tube flash with bottom of the stopper. Pass a rapid stream of hydrogen sulphide through the solution rapidly for half an hour at room temperature. Allow the precipitate to settle for 15 min. Then filter through Whatman No. 42 Filter Paper. Wash the precipitate with water containing a little hydrogen sulphide. Check the washing for complete hardness removal.

A-1.2.3 Dissolve the precipitate in 30 to 40 ml of hydrochloric acid (1:1) and wash the filter paper with a little water. Boil the solution to remove hydrogen sulphide (test the vapour with moist lead acetate paper.) When hydrogen sulphide is completely removed, cool and neutralize with sodium hydroxide using methyl red indicator. Make it up to 500 ml in a volumetric flask.

A-1.2.4 Pipette 50 ml of the made up solution into a conical flask. Add 5 to 6 ml of ammonia buffer solution and 8 to 10 drops of eriochrome black T indicator. Titrate it with EDTA solution (0.01 M). Note the volume of EDTA consumed as V_2.

A-1.3 Calculation

$$\text{Zinc (as Zn), percent by mass} = \frac{V_2 \times M_1 \times 10}{M_2}$$

where

V_2 = volume of EDTA solution (0.01 M) consumed;

M_1 = mass in mg of zinc per ml EDTA solution (0.01 M); and

M_2 = mass in g of sample taken for test under **A-1.2.2**.

Method 2

A-2 MODIFIED ETHYLENE DIAMINE TETRAACETATE (EDTA) METHODS

A-2.1 Reagents

A-2.1.1 *Disodium Ethylene Diamine Tetraacetate (EDTA)*

Dissolve 3.72 g of disodium ethylene diamine tetra-acetate dihydrate in distilled water and make up the volume to 1 litre in a 1 litre volumetric flask.

A-2.1.2 *Standard Zinc Solution*

Weigh about 1.0 g of zinc metal reagent grade accurately. Dissolve in 20 ml of hydrochloric acid (1:1), keep it for few hours and allow it to dissolve completely. Make up the volume of the solution to exactly 1 000 ml.

A-2.1.3 *Ammonium Hydroxide* — 20 percent (*m/m*).

A-2.1.4 *Ammonium Chloride* — AR grade.

A-2.1.5 *Sodium Cyanide* — AR grade.

A-2.1.6 *Sodium Chloride* — AR grade.

NOTE — Sodium cyanide is very poisonous. It should be used with extreme care.

A-2.1.7 *Eriochrome Black T Indicator Mixture*

Mix thoroughly 1 g of eriochrome black T indicator with 100 g of sodium chloride.

A-2.1.8 *Formaldehyde-Acetic Acid Solution (4 Percent)*

Dissolve 100 ml of formaldehyde 37-40 (*m/v)* percent in about 100 ml of distilled water. Add 40 ml glacial acetic acid and make volume to 1 litre with distilled water.

A-2.1.9 *Hydroxyiamine Hydrochloride* — AR grade.

A-2.2 Procedure

A-2.2.1 *Standardization of EDTA Solution*

Take 10 ml standard zinc solution. Add about 0.1 g of ammonium chloride and 30 ml of ammonium hydroxide solution (20 percent). Dilute it by adding about 30 ml distilled water. Add a pinch of eriochrome black T indicator mixture. It will give red colour. Titrate it with EDTA solution to obtain clear blue end point. Note the volume of EDTA used as V_1 ml.

A-2.2.2 *Estimation of Zinc in samples*

Weigh accurately 1.0 g of a given zinc sulphate sample and dissolve it in 100 ml of distilled water in a volumetric flask. Take 10 ml of aliquot in beaker. Add 0.1 g of hydroxylamine hydrochloride and 0.1 g of ammonium chloride. Cautiously add small quantity of sodium cyanide. White precipitate will appear. Continue adding sodium cyanide till white precipitate disappears while swirling the beaker with hand. Add about 0.5 g excess of sodium cyanide. Dilute it by adding about 30 ml of ammonium hydroxide (20 percent) and add about 30 ml of distilled water.

Add a pinch of eriochrome black T indicator mixture. It will give red colour. Titrate with EDTA solution till there is sharp change to violet colour. Note the volume of EDTA used as V_2 ml. Add 20 ml of formaldehyde-acetic acid solution to above titrated solution and mix well. Red colour will reappear. Titrate it with EDTA solution to get blue end point without red tinge. Note the volume of EDTA used in second titration as V_3 ml.

A-2.3 Calculation

$$\text{Zinc (as Zn), percent by mass} = \frac{10 \times V_3 \times M}{V_1}$$

where

M = mass in g of piece of zinc metal taken for preparation of standard zinc solution,

V_1 = volume of EDTA solution (in ml) used for 10 ml of standard zinc solution, and

V_3 = volume of EDTA solution (in ml) used for second titration.

Method 3

A-3 ABSORPTION SPECTROPHOTOMETRIC METHOD

A-3.1 Reagents

Unless specified otherwise, pure chemicals and glass distilled or demineralized water shall be used in tests.

NOTES

1 'Pure chemicals' shall means chemicals that do not contain impurities which affect the results of analysis.

2 'Demineralized water' means the water obtained after passing distilled water through a cation and anion exchange resins or a combined cation-anion exchange resin.

A-3.1.1 *Standard Zinc Solution*

Weigh 0.439 g of zinc sulphate ($ZnSO_4.7H_2O$) AR grade on a clear watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with glass distilled or demineralized

water. Add one ml of 10 percent sulphuric acid (AR grade) and make the volume up to the mark. Stopper the flask and shake the solution well. This is 100 ppm zinc solution hereinafter called Standard A. This solution should be stored in a clean bottle for further use. Dilute 10 ml of 100 ppm solution of Zinc (Standard A) to 100 ml to get 10 ppm standard Zinc solution designated as Standard B.

A-3.1.2 Dilute 1 ml of 10 percent Sulphuric acid to 1 litre with glass distilled or mineralized water and adjust the *p*H to 2.5 with a *p*H meter using H_2SO_4 or NaOH. This solution is called acidified water and 5 to 10 litre of this solution should be prepared at a time.

A-3.2 Preparation of Working Standard

Pipette the following volume of Standard B in 50 ml numbered volumetric flasks and make the acidified water (*see* Table 2).

A-3.3 Procedure

A-3.3.1 *Preparation of Zinc Sulphate Fertilizer Samples*

Weigh 0.25 g of the material on a clean watch glass and transfer it to one litre volumetric flask thorough the funnel giving repeated washings with glass distilled water and dissolve the material by shaking well. Then make the volume up to mark with glass distilled water and shake well.

Take 5 ml of the prepared solution in 250 ml volumetric flask and make the volume with acidified water. Shake the solution well and filter through Whatman No. 42 filter paper in dry clean flask. The flasks should be rinsed with a 10 to 15 ml of the filtrate and then continue filtration.

A-3.3.2 *Flaming the Solutions*

Flame the standards and the filtered samples for atomic absorption spectrophotometer at a wavelength of 213.8 nm (Zn line of the instrument).

A-3.4 Calculation

Prepare a standard curve of known concentrations of zinc solution by plotting the absorbance values of Y axis. Calculate the percentage zinc fertilizer by multiplying zinc concentration value calculated from standard curve by 20.

Example:

Mass of the fertilizer sample	:	0.25 g
Volume made	:	1 000 ml
Further dilution	:	50 times
Reading of the samples from Atomic absorption	:	Y
Corresponding, concentration Value of zinc from standard curve Against Y absorbance	:	X ppm
Percentage zinc in the fertilizer	:	20 (X)

A-3.5 Precautions

a) Weighing must be done on an electric balance;
b) All the glass apparatus to be used should be neutral and washed with dilute hydrochloric acid (1:4) and washed thoroughly with distilled and then with demineralized water;
c) The pipette should be rinsed with the same solution to be measured;
d) The outside of the pipette should be wiped with filter paper after taking out from the solution to be measured;
e) After using the pipette, place them on a clean dry filter paper in order to prevent contamination; and
f) To start filtration, only a few drops should be added first in order to wet the filter paper and then continue further filtration.

Stopper the flasks and shake them well. Prepare the standard in duplicate. The same acidified water should be used for preparing the solution of unknown fertilizer samples. Fresh standards should be prepared every time when a fresh lot of acidified water is prepared.

Table 2 Preparation of Working Standards
(*Clause* A-3.2)

Flask No.	Volume of Standard B Takes, ml	Concentration of Zinc After Making Volume to 50 ml (ppm)
1	0.0	0.0
2	1.0	0.2
3	2.0	0.4
4	3.0	0.6
5	4.0	0.8
6	5.0	1.0
7	7.0	1.4
8	9.0	1.8
9	10.0	2.0

ANNEX B

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF MAGNESIUM

B-1 Two methods have been specified EDTA method and Atomic Absorption Spectroscopic method. The Atomic Absorption Spectroscopic method shall be taken as reference method in case of any dispute.

B-2 EDTA METHOD

B-2.1 Reagents

B-2.1.1 *Dilute Sulphuric Acid* — approximately 5 N.

B-2.1.2 *Dilute Nitric Acid* — approximately 10 percent (*v*/*v*).

B-2.1.3 *Sodium Sulphide Solution* — 10 percent.

B-2.1.4 *Eriochrome Black T indicator* — Dissolve 0.1 g of Eriochrome black T in 25 ml of methyl alcohol.

B-2.1.5 *Diammonium Hydrogen Phosphate* — 10 percent (*v*/*v*).

B-2.1.6 *Ammonium Hydroxide — Ammonium Chloride Buffer Solution*

Mix 350 ml of ammonium hydroxide (20 percent, *m*/*m*) with 34 g of ammonium chloride. Dilute with water and make up the volume to 1 000 ml. (The *p*H of the solution should be not more than 10.)

B-2.1.7 *Standard Magnesium Solution* — 0.01 M.

B-2.1.7.1 Weigh 2.464 0 g of magnesium sulphate ($MgSO_4,7H_2O$) and dissolve it in water. Make up the volume to one litre.

B-2.1.8 *Ethylenediamine Tetraacetate (EDTA) Solution*

Dissolve 3.72 g of disodium ethylenediamine tetraceetate dehydrate in water and make up the volume to one litre.

B-2.1.8.1 *Standardization of EDTA solution*

Take 10 ml of standard magnesium solution in a conical flask. Add 20 ml of water, 1ml of Eriochrome black T indicator and 25 ml of ammonium hydroxide ammonium chloride buffer solution. Heat to 40 to 50°C and then titrate with EDTA solution, maintaining the temperature between 40 and 50°C until the colour changes from wine red to distinct blue. Calculate the molarity of EDTA solution as follows:

$$\text{Molarity of EDTA solution} = \frac{10\,M_1}{V_1}$$

where

M_1 = molarity of standard magnesium solution, and

V_1 = volume in ml of EDTA solution used for titration.

B-2.2 Procedure

B-2.2.1 Weigh accurately about 5 g of the sample, dissolve in water and add 1 ml of dilute sulphuric acid. Filter the solution and make up to 250 ml with water in a volumetric flask. Take 50 ml of the above solution in a beaker, heat, pass hydrogen sulphide gas or add sodium sulphide solution and ensure complete precipitation. Filter, hot and keep the filtrate for the determination of magnesium as given in **B-2.2.2.** Boil the residue with dilute nitric acid and filter if necessary. To the filtered solution add dilute sulphuric acid, evaporate, dilute and filter. Use the filtrate for the determination of copper and the residue for the determination of lead.

B-2.2.2 Take the filtrate obtained in **B-2.2.1** after precipitation of sulphides add a few drops of concentrated nitric acid, boil and cool and then add solid ammonium chloride (about 2 g), boil and cool, add ammonium hydroxide till strong smell of ammonia comes and filter the precipitate through sintered crucible. Take the filtrate and add dilute sulphuric acid till the solution is acidic (test with methyl red), heat the solution to boil and add excess of diammonium hydrogen phosphate with continuous stirring. Add 10 percent ammonia solution with continuous stirring till the solution is just alkaline (test with methyl red); white precipitate of zinc ammonium phosphate will be formed (the optimum *p*H is 6 to 7). Allow it to stand for 3 to 4 h, then filter through filter paper (Whatman No. 40 or equivalent). Collect the filtrate in a volumetric flask and make up the volume (say to 100 ml), take a suitable aliquot (say 10 ml) for the determination of magnesium. Add 20 ml of water, 1 ml of eriochrome black T indicator and 20 ml of magnesium. Add 20 ml of water, 1 ml of eriochrome black T indicator and 20 ml of ammonium hydroxide-ammonium chloride buffer solution. Heat to 40 to 50°C and titrate with standard EDTA solution, maintaining the temperature between 40 to 50°C until the colour changes from wine red to distinct blue.

B-2.3 Calculation

1 ml of 0.01 M EDTA = 0.2432 mg of 'Mg'

$$\text{Magnesium (as Mg), percent by mass} = \frac{V \times 0.243\,2}{5}$$

where

V = volume of 0.01 M EDTA solution used for titration.

B-2.3.1 The calculation factor 5 is derived presuming that 5 g of material is taken for test in **B-2.2.1** and the filtrate obtained in **B-2.2.2** is 100 ml out of which 10 ml is titrated.

B-3 ATOMIC ABSORPTION SPECTROSCOPIC METHOD

B-3.1 Reagents

B.3.1.1 *Strontium Chloride*

Dissolve 7.5 g of strontium chloride ($SrCl_2.6H_2O$) in one litre of glass distilled water.

B-3.1.2 *Standard Magnesium Solution*

Weigh 0.507 g of magnesium sulphate ($MgSO_4.7H_2O$) on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with glass distilled or demineralized water. This is 50 ppm Mg solution. Dilute 10 ml of 50 ppm solution of Mg to 100 ml to get 5 ppm standard Mg solution.

B-3.1.3 *Preparation of Working Standards*

Pipette the following volume of 5 ppm standard Mg solution in 50 ml numbered volumetric flasks. Add 10 ml of strontium chloride solution to each flask and make up the volume to 50 ml.

Stopper the flask and shake them well. Prepare fresh standards every fortnight.

B-3.2 Procedure

B-3.2.1 Pipette 20 ml of a solution which was prepared for the determination of zinc by dissolving 0.25 g of the fertilizer sample in one litre flask. Add 10 ml of strontium chloride. Make up the volume to 50 ml.

B-3.2.2 Flame the standards and the samples on atomic absorption spectroscopic photometer at the wavelength of 285.5 mμ (Mg line of the Instrument).

B-3.3 Calculations

Prepare a standard curve of known concentrations of Mg solutions by plotting the absorbance value on Y-axis against their respective concentration values on X-axis. Percentage magnesium in the zinc fertilizer will correspond to the concentration values calculated from the standard curve.

Example:

Mass of the fertilizer	=	0.25 g
Volume made	=	1 000 ml
Further dilution	=	2.5 times
Reading of the sample from absorption spectrophotometer	=	Y
Corresponding concentration of Mg from standard curve against Y absorbance	=	X
Percentage magnesium in the fertilizer	=	X

Flask No.	*Volume of 5 ppm Mg Solution taken (ml)*	*Volume of Strontinum Chloride Added (ml)*	*Concentration of Magnesium after Making the Volume to 50 ml (ppm)*
1	0.0	10.0	0.0
2	2.0	10.0	0.2
3	4.0	10.0	0.4
4	6.0	10.0	0.6
5	8.0	10.0	0.8
6	10.0	10.0	1.0

ANNEX C

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF COPPER

C-1 Two methods have been specified chemical method [Diethyldithiocarbamate method or biquinoline method) or Atomic absorption spectroscopic method shall be reference method in case of any dispute.

C-2 CHEMICAL METHOD

C-2.1 Procedure

Make up the filtrate received in **B-2.2.1** for the determination of copper, to 200 ml with water in a volumetric flask. Take a suitable aliquot of the solution containing not more than 0.05 mg of copper. Determine copper by diethyl dithiocarbamate method or by biquinoline method as prescribed in IS 7212.

C-3 ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

C-3.1 Reagents

C-3.1.1 *Standard Copper Solution*

Weigh 0.196 5 g of copper sulphate ($CuSO_4.5H_2O$) on clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and the funnel with glass distilled water. Add one ml of 10 percent sulphuric acid and make up the volume up to the mark. Stopper the flask and shake the solution well. This is 50 ppm Cu solution and should be stored in a clean bottle for further use. Dilute 10 ml of 50 ppm solution of copper to 100 ml to get 5 ppm standard copper solution.

C-3.1.2 Glass distilled or mineralized acidified water of *p*H 2.5 ± 0.5.

C-3.1.3 *Preparation of Working Standards*

Pipette the following volume of 5ppm standard copper solution in 50 ml numbered volumetric flasks and make the volume with acidified water.

Stopper the flasks and shake them well. Prepare fresh standards every fortnight.

C-3.2 Procedure

C-3.2.1 The solution which was prepared for the determination of zinc by dissolving 0.25 g of the fertilizer sample in one litre flask should be used for the determination of copper.

C-3.2.2 Flame the standards and the samples on an atomic absorption spectrophotometer at a wavelength of 324.8 mμ (Cu line of the instrument).

C-3.3 Calculations

Prepare the standard curve of known concentrations of copper solutions by plotting the absorbance values on Y-axis against their respective concentration values on X-axis. Calculate the percentage copper in the zinc fertilizer by multiplying the copper concentration value calculated from the standard curve by 0.4.

Example:

Mass of the fertilizer sample = 125

Volume made = 1 000 ml

Reading of the sample from atomic absorption spectrophotometer = Y

Corresponding concentration of copper X ppm from standard curve against Y absorbance

Percentage copper in the fertilizer = 0.4 X

Flask No.	*Volume of 5 ppm Standards Cu Solution taken* (ml)	*Concentration of Copper after Making the Volume to 50 ml (ppm)*
1	0.0	0.0
2	2.0	0.2
3	4.0	0.4
4	6.0	0.6
5	8.0	0.8
6	10.0	1.0

ANNEX D

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF LEAD

D-1 Two methods have been specified dithiozone method and atomic absorption spectrophotometric method. The atomic absorption spectrophotometric method shall be taken as reference method.

D-2 DITHIOZONE METHOD

D-2.1 Procedure

Dissolve the residue received in **B-2.2.1** for determination of lead in dilute nitric acid, and make up to a known volume with water in a volumetric flask. Take a suitable aliquot of the solution and determine lead by the calorimetric method using dithiozone as prescribed in IS 7017.

D-3 ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

D-3.1 Reagents

D-3.1.1 *Standard Lead Solution*

Weigh 0.159 9 g of lead nitrate [$Pb(NO_3)_2$] on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with glass distilled or demineralized water. Add 10 ml of concentrated distilled nitric acid and make the volume up to the mark. Stopper the flask and shake the solution well. This is 100 ppm lead solution and should be stored in a clean bottle for further use. Dilute 10 ml of 100 ppm solution of lead to 100 ml with 1 percent nitric acid solution to get 10 ppm standard lead solution.

D-3.1.2 *One Percent Nitric Acid Solution*

Dilute 10 ml of concentrated distilled nitric acid to one litre with glass distilled water.

D-3.1.3 *Twenty Percent Zinc Sulphate Solution*

Weigh 20 g of zinc sulphate ($ZnSO_4.7H_2O$) and dilute to 100 ml with 1 percent nitric acid solution.

D-3.1.4 *Preparation of Working Standards*

Pipette the following volume of 10 ppm standard lead solution in 50 ml numbered volumetric flasks. Add 5 ml of 20 percent zinc sulphate solution to each flask and make the volume with 1 percent nitric acid solution.

Stopper the flask and shake them well.

D-3.2 Procedure

D-3.2.1 *Preparation of Zinc Sulphate Fertilizer Samples*

Weigh 1 g of the material on a clean watch glass and transfer to 50 ml volumetric flask through the funnel giving washings with 1 percent nitric acid solution. Dissolve the material and make up the volume with 1 percent nitric acid solution. Sample should be prepared in duplicate.

D-3.2.2 *Flaming the Solutions*

Flame the standards and the samples on atomic absorption spectrophotometer at a wavelength of 217 mμ (Lead line of the instrument).

D-3.3 Calculations

Prepare a standard curve of known concentrations of lead solutions the abosorbance values on Y-axis against their respective lead concentration on X-axis. Calculate the percentage lead in zinc fertilizer by multiplying lead concentration value calculated from standard curve by 0.005.

Flask No.	*Volume of 10 ppm Lead Solution taken (ml)*	*Volume of 20 percent Zinc Sulphate Solution Added (ml)*	*Concentration of Lead after Making the Volume to 50 ml (ppm)*
1	0.0	5.0	0.0
2	2.0	5.0	0.4
3	4.0	5.0	0.8
4	6.0	5.0	1.2
5	8.0	5.0	1.6
6	10.0	5.0	2.0

ANNEX E

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF *p*H

E-1 APPARATUS

E-1.1 *p*H Meter — Use a standard laboratory *p*H meter (*see* IS 2711).

E-2 PROCEDURE

Dissolve 5 g of the material in freshly boiled and cooled water, dilute to 100 ml and mix. Determine the *p*H value of the solution with a *p*H meter. Repeat the test and record the mean *p*H value.

ANNEX F

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF MATTER INSOLUBLE IN WATER

F-1 REAGENT

F-1.1 Dilute Sulphate Acid — 10 percent.

F-1.2 Procedure

Dissolve 25.0 g of the material in 125 ml of water and add 1 ml of dilute sulphuric acid. Heat the solution to boiling. Filter through a weighted and prepared Gooch crucible or sintered glass crucible (G No. 4) and wash the residue thoroughly with hot water. Dry the crucible at 110 ± 5°C to constant mass.

F-1.3 Calculation

$$\text{Matter insoluble in water} = 4A$$

where

A = mass in g of the residue.

ANNEX G

[*Table* 1, *Sl No.* (viii)]

DETERMINATION OF CADMIUM

G-1 APPARATUS

G-1.1 Beaker, 250 ml capacity.

G-1.2 Volumetric Flask, 100 ml and 1 000 ml capacity.

G-1.3 *p*H Meter

G-1.4 Atomic Absorption Spectrophotometer, with rich air-acetylene flame.

G-2 REAGENTS

G-2.1 Standard Cadmium Solution — Weigh out 1 g of pure cadmium metal and transfer it to a 250 ml beaker. Add 50 ml of water and 10 ml of concentrated nitric acid to dissolve the metal completely. Transfer the cadmium solution to a one litre volumetric flask with necessary washing. Make up the volume up to the mark. Shake well. This is a 1 000 ppm solution of

cadmium, (hereinafter called standard A). Dilute 1 ml of standard A to 100 ml in a volumetric flask. This is a 10 ppm solution of cadmium (hereinafter called Standard B).

G-2.2 Glass Distilled Water of *p*H 2.5 ± 0.5 — Dilute 1 ml of 10 percent suphuric acid to one litre with glass distilled water and adjust the *p*H to 2.5 ± 0.5 with a *p*H meter using sulphuric acid or sodium hydroxide solution. The water so obtained is called acidified water.

G-2.3 Preparation of Working Standards — Pipette out the following volume of standard B in 100 ml of numbered volumetric flask and make up the volume with acidified water. Stopper the flask and shake them well. The same acidified water should be used for the preparation of the sample solution. Fresh standards should be prepared and used every time.

G-3 PROCEDURE

G-3.1 Preparation of Sample Solution

Weigh 2 g of zinc sulphate and transfer it to a 100 ml volumetric flask giving repeated washings with acidified water. Dissolve the material by shaking well, make up the volume and mix thoroughly. Filter a portion if necessary. For higher concentration of cadmium, adjust the weight and dilution such that the absorbance of final flaming solution is not more than a 2 ppm solution of cadmium.

G-3.2 Aspirate the standards as well as the sample solution in an atomic absorption spectrophotometer at a wavelength of 228.8 mμ using air acetylene flame and note the corresponding absorbance value for each solution.

G-4 CALCULATION

Draw a graph using concentration (ppm) as the X-axis and absorbance as the Y-axis. Determine the concentration of cadmium in ppm in the sample solution from the graph.

Concentration of Cadmium (as Cd), in ppm $= \frac{c \times d_f}{W}$

where

c = concentration of final sample solution, in ppm;

W = weight of sample, in g; and

d_f = dilution factor

Flask No.	*Volume of Standard B taken (in ml)*	*Concentration of Cadmium After Making Volume to 100 ml (in ppm)*
1	0.0	0/0
2	2.0	0.2
3	4.0	0.4
4	8.0	0.8
5	12.0	1.2
6	16.0	1.6
7	20.0	2.0

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc No.: FAD 07 (2827).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.org.in

Regional Offices: *Telephones*

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 — 2323 7617, 2323 3841

Eastern : 1/14 C.I.T. Scheme VII M, V. I. P. Road, Kankurgachi, KOLKATA 700054 — 2337 8499, 2337 8561, 2337 8626, 2337 9120

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, CHANDIGARH 160019 — 26 50206, 265 0290

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113 — 2254 1216, 2254 1442, 2254 2519, 2254 2315

Western : Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400093 — 2832 9295, 2832 7858, 2832 7891, 2832 7892

Branches: AHMEDABAD. BENGALURU. BHOPAL. BHUBANESWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi